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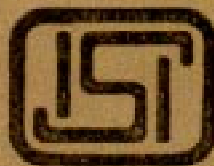
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Indian Standard

METHOD FOR DETERMINATION OF
RELATIVE REDUCIBILITY OF IRON OXIDES:
LUMP ORES, SINTER AND PELLETS

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INDIAN STANDARDS INSTITUTION

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Indian Standard

METHOD FOR DETERMINATION OF RELATIVE REDUCIBILITY OF IRON OXIDES: LUMP ORES, SINTER AND PELLETS

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Indian Standard

METHOD FOR DETERMINATION OF RELATIVE REDUCIBILITY OF IRON OXIDES: LUMP ORES, SINTER AND PELLETS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 30 May 1985, after the draft finalized by the Ores and Raw Materials Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 The reducibility test helps in choosing the appropriate iron oxide feed, while the relative reducibility test aids in a more meaningful comparison of different oxide feeds. This standard has been prepared to establish uniform practice for the determination of relative reducibility of iron oxides: lump ores, sinters and pellets.

0.3 This standard differs from the existing standard (IS : 8167-1976*) on reducibility in so far as it expresses the total weight loss at the end of 3 hours instead of $\frac{DR}{Dt}$ at 40 percent reduction, where the time is not specified.

0.4 In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960†.

1. SCOPE

1.1 This standard prescribes the method for the determination of relative reducibility of iron oxides; lump ores, sinters and pellets. The test is to be conducted under specific conditions, for example, isothermal reduction, reduction in a fixed bed, reduction by means of carbon monoxide and nitrogen mixture, and a sample having a specified size range.

2. TERMINOLOGY

2.1 For the purpose of this standard, the following definitions shall apply.

*Method for determination of reducibility of iron ore sinters.

†Rules for rounding off numerical values (revised).

2.2 Reducibility — A measure of the ease with which oxygen combined with iron can be removed from natural or processed iron ores with a reducing gas.

2.3 Relative Reducibility — The final degree of reduction attained after a prescribed period of time and with other specified condition.

3. PRINCIPLE AND SUMMARY OF TEST

3.1 Relative reducibility is determined in a specified range in a fixed bed at fixed temperature using a reducing gas mixture consisting of $\text{CO} + \text{N}_2$, thus determining the percent oxygen removal in a fixed time. The sample is placed in a reduction tube, which is inserted into a furnace and connected to a weighing device. The sample is heated to the specified temperature while inert gas (nitrogen) is flowing through it. The sample is continuously weighed and the loss of oxygen is determined by knowing the weight of sample before and after the reduction. The cooling of the sample is also done in a nitrogen atmosphere.

4. SAMPLES

4.1 The relative reducibility test sample may be taken from the samples collected for other physical tests, for example, shatter index test and tumbler test.

4.2 The total mass of the relative reducibility test sample should be approximately 500 g.

4.3 The test sample for lump ore, sinter or pellets shall be in the range of $-12.5 \text{ mm} + 10 \text{ mm}$. In all cases, alternative size ranges for lump ore, sinter and pellets may also be used by agreement between the parties concerned.

4.4 A portion of the sample should be set aside for the purpose of chemical analysis, for the determination of total iron amount of oxygen combined with iron and loss on ignition. These constituents shall be determined by the methods specified in IS : 1493-1959* and IS : 1493 (Part 1)-1981† or by any other instrumental methods.

5. TEST CONDITION

5.1 Gas volumes and flow rates in this test shall be measured at standard temperature and pressure.

5.2 The reducing gas for the entire test shall be a mixture of $\text{CO } 30 \pm 1 \text{ percent (V/V)}$, and $\text{N}_2 70 \pm 1 \text{ percent (V/V)}$ and shall be passed through the drying tower or U-tube containing blue silica gel following removal of CO_2 , O_2 and other impurities.

*Methods of chemical analysis of iron ores.

†Methods of chemical analysis of iron ores : Part 1 Determination of common constituents.

5.3 The reducing gas before entering the basket holding the sample shall be preheated to $900 \pm 10^{\circ}\text{C}$. The preheating shall be attained in the same reactor.

5.4 The reduction gas flow shall be maintained at 15 l/min at standard temperature and pressure during the entire test period.

5.5 The temperature of the reduction sample shall be maintained at $900 \pm 10^{\circ}\text{C}$ throughout the entire bed of the sample during the test period.

5.6 The test shall be performed for 180 min. The sample shall then be cooled under a flow of nitrogen.

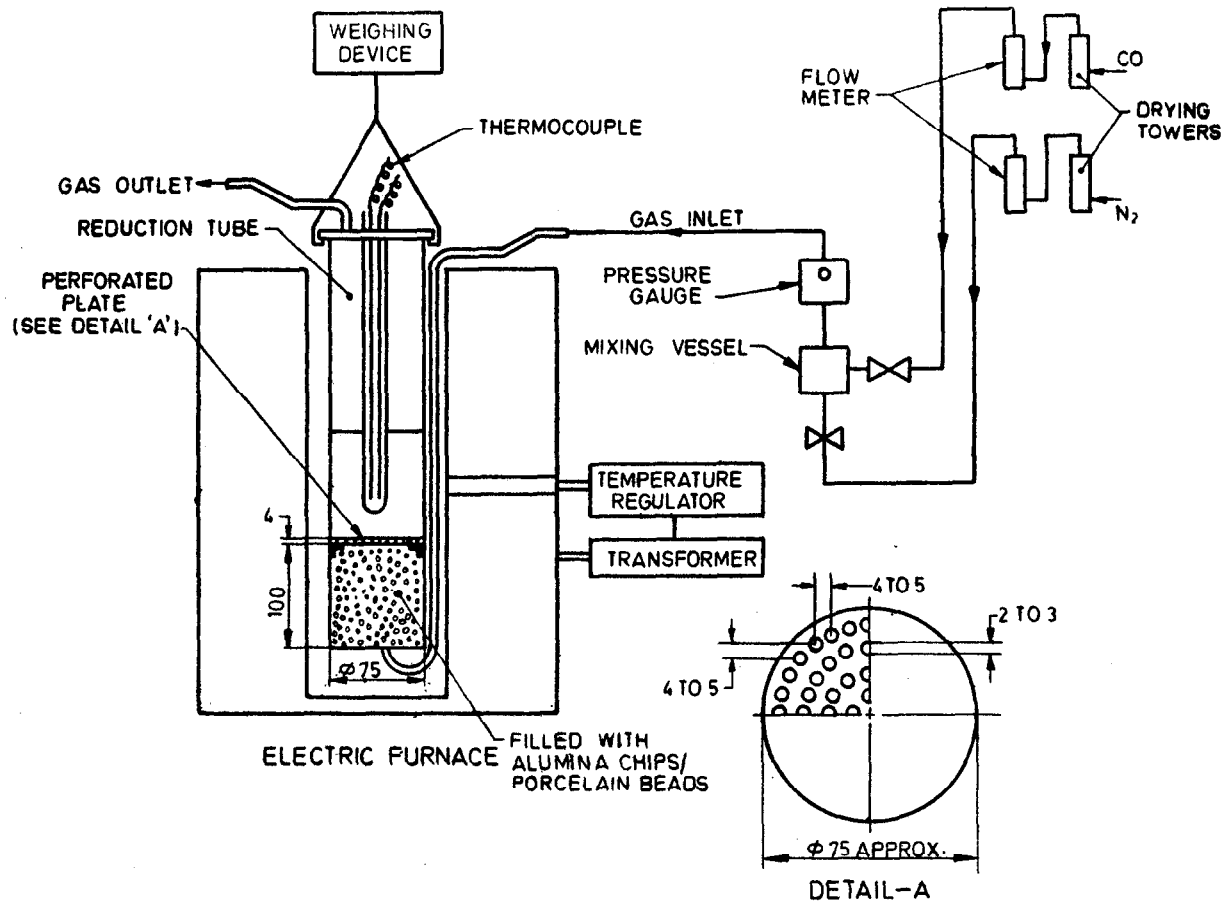
6. APPARATUS

6.1 The apparatus shall consist of the following:

- a) A system to supply and regulate the flow of gases,
- b) A reduction tube assembly,
- c) A continuous weighing device to determine the oxygen loss at any time during the test,
- d) An electrically heated furnace to heat the test portion to the specified temperature, and
- e) Test sieves.

6.2 The reduction vessel consists of a tube with suitable top and bottom flanges to make it leakproof. The heating zone shall be so adjusted that the lower part of the zone may be utilized for heating the reducing gas to the desired temperature. The reduction tube is suspended in an electrically heated furnace on a balance which indicates the variation of the mass of the sample continuously during reduction. Nitrogen and reducing gas taken from gas cylinders, or other sources, are expanded, dried and introduced into the reduction tube. The waste gas is drawn off from the upper part of the tube. Caution shall be exercised to dispose off the waste gas by burning or by some other appropriate means. A calibrated thermocouple, suitably positioned, inside the reduction vessel shall be used to record the temperature. A typical arrangement of apparatus is shown in Fig. 1.

6.3 The reduction tube shall be made of heat resisting non-scaling steel. The height of the reduction tube be so adjusted that the tube is in the constant temperature zone of the furnace. The space between the furnace and the reduction tube shall be sealed. A typical illustration of reduction tube is shown in Fig. 1.



All dimensions in millimetres.

FIG. 1 SCHEMATIC DIAGRAM OF REDUCTION APPARATUS

6.4 Furnace — The furnace shall be capable of attaining a temperature of the sample $900 \pm 10^\circ\text{C}$ in 60 minutes. The entire test sample shall be maintained at this temperature throughout the test period.

6.5 Weighing Device — The sensitivity of the thermogravimetric weighing device, which allows *in situ* measurement of weight loss during reduction, shall be ± 0.5 g and with a suitable load capacity. The initial mass of the test sample (m_0) shall be recorded with this sensitivity.

6.6 Sieves — 10 and 12.5 mm square sieves shall be used.¶

7. PROCEDURE

7.1 A pre-dried sample (dried at a temperature of $150\text{--}200^\circ\text{C}$) of 500 g shall be inserted in the reduction tube and after placing the heat exchange medium under the perforated plate, shall be evened out by tapping. The tube, holding the sample shall then be suspended freely from the thermogravimetric balance without any contact with the furnace walls.

7.2 The air in the tube shall be replaced with nitrogen. The sample shall be heated and while heating, a flow rate of approximately 5 litre per minute at NTP of nitrogen shall be passed through the sample. After the temperature of the sample reaches 900°C , the flow rate of nitrogen shall be increased to 15 litres per minute at NTP during the soaking period. The sample shall be then allowed to soak at this temperature for 30 minutes minimum. The mass of the sample immediately before starting reduction shall be weighed and recorded as m_1 .

7.3 The mass shall be recorded every 3 minutes for the first 15 minutes and thereafter at 10 minutes intervals.

7.4 The test shall be performed for 180 minutes. The sample shall then be cooled under a flow of purified nitrogen. The mass of the sample shall then be taken and recorded as m_2 .

8. EXPRESSION OF RESULTS

8.1 Calculation of Relative Reducibility (Degree of Reduction)

8.1.1 The total amount of oxygen combined with iron shall be calculated from the chemical composition and the sample mass. The degree of reduction attained after 3 hours, referred to as the relative reducibility (final degree of reduction), R_f , expressed as a percentage, shall be calculated from the following formula as derived in Appendix A, to the first decimal place.

$$R_f = \frac{m_1 - m_2}{m_0 (43 W_1 - 11.1 W_2)} \times 100$$

where

- m_0 is initial mass of test sample, in g;
- m_1 is mass of the test sample, immediately before starting the reduction, in g;
- m_2 is mass of the test sample after 3 h of reduction, in g;
- W_1 is the total iron content, expressed as a mass fraction, of the sample prior to the test; and
- W_2 is the ferrous oxide content, expressed as a mass fraction, of the sample prior to the test.

8.1.2 Number of Tests — At least two reduction test shall be carried out. If the difference between both the results of R_f are within 2.5 percent of the mean, the mean may be reported as relative reducibility, expressed in percentage, to the first decimal place. In case the two tests do not agree to this extent, two more tests shall be conducted and this norm fulfilled.

8.1.3 Calculation of Final Results — The relative reducibility, R_f , expressed as percentage, shall be reported as the arithmetic mean of all test results, rounded to the nearest whole number.

9. REPORTING OF RESULT

9.1 The test report shall contain the following information:

- a) reference to this standard;
- b) total iron, metallic iron and oxygen combined with iron (FeO , Fe_3O_4 and Fe_2O_3) of the test sample, after the test;
- c) the whole oxygen removal time curve, if required;
- d) the final degree of reduction, R_f ;
- e) degree of reduction at any time t , R_t ($t < 3\text{h}$), calculated from

$$R_t = \frac{m_1 - m_t}{m_1 - m_2} \times R_f$$

where,

- m_t is the mass of the sample obtained from (c) above at any time t ($t < 3\text{h}$);
- f) the atmospheric pressure at the time of test;
- g) the number of particles in the test sample for calculation of the surface area; and
- h) the height of the sample bed before and after testing.

APPENDIX A

(Clause 8.1)

DERIVATION OF THE FORMULA FOR RELATIVE REDUCIBILITY

A-1. BASIC EQUATION

A-1.1 The equation for R_t given in 8.1 is derived from the basic equation

$$R_t = \frac{\Delta m_t}{\Delta m_3} \times 100 \quad \dots\dots\dots (1)$$

where

m_t is the mass loss, in grams, of oxygen during 3 h of reduction;
 m_3 is the mass, in grams, of oxygen combined with iron before reduction.

A-2. DERIVATION OF WORKING EQUATION

A-2.1 The iron oxides contained in the sample are considered to be haematite (Fe_2O_3), magnetite ($\text{FeO} \cdot \text{Fe}_2\text{O}_3$) and ferrous oxide (FeO). The total mass of oxygen, m_3 , in equation (1) may be obtained from the masses of Fe_2O_3 and FeO in the test sample before reduction. Therefore, m_3 is given by equation (2), after the total iron content, W_1 , and the ferrous oxide content, W_2 , of the test sample have been determined in accordance with IS : 1493-1959*

$$m_3 = m_4 + m_5 = m_o \left(W_3 \frac{3A_o}{2A_{\text{Fe}}} + W_2 \frac{A_o}{M} \right) \quad \dots\dots\dots (2)$$

where

m_4 is the mass, in grams, of oxygen in Fe_2O_3 ;
 m_5 is the mass, in grams, of oxygen in FeO ;
 m_o and W_2 have the same meanings as in 8.1;
 W_3 is the iron content, expressed as a mass fraction, in Fe_2O_3 ;
 A_o is the relative atomic mass of oxygen, 16.00;
 A_{Fe} is the relative atomic mass of iron, 55.85;
 M is the relative molecular mass of ferrous oxide, 71.85;

*Methods of chemical analysis of iron ores.

Noting that

$$\Delta m_t = m_1 - m_2$$

$$W_3 = W_1 - \frac{A_{Fe}}{M} W_2$$

where m_1 , m_2 , W_1 and W_2 have the same meanings as in 8.1, and substituting m_3 , from equation (2) into equation (1), the final degree of reduction, R_f , expressed as a percentage, is given by the equation:

$$\begin{aligned} R_f &= \frac{(m_1 - m_2) \times 100}{m_o \left[\left(W_1 - \frac{A_{Fe}}{M} W_2 \right) \frac{3}{2} \frac{A_o}{A_{Fe}} + \frac{A_o}{M} W_2 \right]} \\ &= \frac{m_1 - m_2}{m_o \left[\left(W_1 - \frac{55.85}{71.85} W_2 \right) \frac{48.00}{111.70} + \frac{16.00}{71.85} W_2 \right]} \times 100 \\ &= \frac{m_1 - m_2}{m_o (0.430 W_1 - 0.111 W_2)} \times 100 \end{aligned}$$